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RELEASE MECHANISMS OF ORGANOTIN TOXICANTS FROM COATING SURFACES--ETC(U)

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⁶ RELEASE MECHANISMS OF ORGANOTIN TOXICANTS FROM COATING SURFACES:
A LEACHING MODEL FOR ANTIFOULING COATINGS.

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by

¹⁰ Charles P. Monaghan / Vasant H. Kulkarni and Mary L. Good

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ABSTRACT

Fouling of a ship bottom or a marine installation is usually combated by using a coating which contains an antifouling toxicant. The toxicant leaches out of the coating and prevents organisms from settling on the protected surface. An empirical diffusion model has been developed which describes the leaching process of organotin toxicants out of antifouling coatings. From this model the concentration of organotin in the leachate, c , is given by

$$c = c_s - c_s \exp\left(-\alpha \frac{A_o}{V} t\right)$$

where c_s is the concentration of organotin at the coating surface, A_o is the measured surface area of the coating plane, V is the volume of seawater in the experimental container, and t is time. The empirical parameter, α , is characteristic for a particular coating at a specified temperature and water flow condition at the coating surface.

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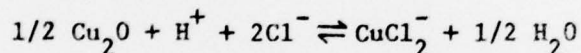
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Fouling of ship hulls and marine installations by vegetable and/or animal organisms is a well-known, worldwide problem which results in significant economic loss due to structural damage or fuel requirements (1,2,3). Thus any program for fuel economy and preservation of marine structures by the Navy, the shipping industry, or the fishing fleet must contain the development of effective antifoulant procedures as a high priority. The only practical solution to the fouling problem through the years has been the utilization of paints and coatings having antifoulant activity. Most of the successful coatings have been paint formulations containing a toxic component. The most widely used toxicant has been cuprous oxide although various organometallic compounds of arsenic, mercury, lead and tin have been used. Studies have indicated that the organotin antifoulants show particular promise, since they exhibit control over a variety of fouling organisms for relatively long periods of time and they do not promote corrosion when applied over conductive substrates (4,5). However, if these organotin containing coatings find widespread application in the marine industry and in naval operations, their long term environmental impact on harbors and shipyards will become most important. In addition, the need for laboratory testing techniques for coating evaluation and comparison will be critical. Thus the development of laboratory procedures for the chemical speciation of the toxicant released to the environment and methods for evaluating the leaching parameters becomes an area of fertile research. This paper will report our initial efforts to develop in detail a leaching model for the loss of organotin toxicants from conventional antifouling coatings. Work on the speciation of the released toxicant moieties will be presented in a subsequent paper.

For an antifouling paint to be effective, the toxicant must leach from the coating at a rate high enough to repel or kill organisms at the coating surface but not so high as to cause rapid depletion and early failure of the coating. De la Court and de Vries (6,7) and Marson (8,9) have outlined those parameters

which are necessary to describe the leaching of toxicant from an insoluble matrix containing cuprous oxide (Cu_2O). The models developed by these authors show considerable insight into the leaching process.

Coatings in which the toxicant particles and soluble pigment particles are dispersed throughout the coating matrix in such a manner that the particles are always in contact are called "continuous contact coatings". In these coatings, it is assumed that when the particles dissolve, pores are developed through which the toxicant diffuses to the surface of the coating and the porous exhausted matrix remains intact after the particles have dissolved. Several steps are required to describe the leaching process. The solvent diffuses through the exhausted matrix to the leaching zone where it dissolves the toxicant or soluble pigment, leaving a pore. The solvated toxicant molecule diffuses through the exhausted matrix to the surface and then diffuses through the quiescent layer into the bulk of the solution (sea or harbor water). Cuprous oxide dissolves in sea water to establish a reaction equilibrium as follows:



The rate determining steps for toxicant leaching are considered to be the diffusion of CuCl_2^- through the exhausted matrix and the subsequent diffusion of CuCl_2^- through the quiescent layer. If these assumptions are correct, a concentration approaching saturation will develop in the leaching zone. A stationary state will be established where the concentration of the toxicant is constant throughout any cross section of the exhausted matrix and throughout the quiescent layer. Under these conditions the flux of toxicant through a unit cross section is the same in both the exhausted matrix and the quiescent layer. It is evident from the formulation of this model that a detailed study of the matrix is necessary to characterize the leaching process.

There are several points where coatings containing organotin compounds differ from those containing cuprous oxide. These differences complicate the study of the leach rates and makes examination of the surface mandatory. The most effective organotin toxicants, of general formula R_3SnX , exist either as solids or as liquids. For conventional coatings where the toxicant is physically mixed with the paint matrix in a common solvent, the liquid organotins would be expected to disperse homogeneously throughout the coating; whereas, the solid materials may disperse as fine particles forming a heterogeneous mixture. The usual pigments, carbon, ferric oxide and titanium dioxide, are insoluble in most coating formulations and are also dispersed as fine particles. In successful organotin containing coatings, the percentage of toxicant is about ten percent or less as contrasted to the forty to fifty percent toxicant composition commonly used in cuprous oxide coatings. Thus the "continuous contact" leaching

models are not appropriate for these materials. It has been proposed (10, 11, 12, 13) that some of the matrix must dissolve for leaching to occur in a coating containing organometallic toxicants. To evaluate this proposition the coating surface must be examined in detail to characterize the aging mechanism (dissolution of coating matrix, cracking, sloughing off of exhausted matrix, etc.) and/or the leach solution must be analyzed for dissolved matrix. It has also been proposed (10) that the leachate species for all of the coatings containing R_3SnX is the same molecule, namely R_3SnOH , and that the leaching rate depends not only on the matrix, but also on the physical state of the toxicant in the coating.

The model developed below has been designed with the view that a practical goal of any leaching model is to predict the useful lifetime of an antifouling coating with a minimum amount of laboratory and field testing. Thus the model presented here is a general empirical approach to the leaching problem with little emphasis on microscopic detail. Laboratory tests can be performed and the model can be used to determine the amount of organotin remaining in the matrix for a given set of experimental conditions. A reasonable extension of the model is the prediction of minimum coating lifetimes by defining the extreme conditions that a coating is likely to encounter and then designing laboratory testing procedures which simulate those conditions.

Details Of The Leaching Model

At any time during the lifetime of the antifouling coating, toxicant molecules diffuse out of the coating in a direction normal to the coating plane. The molecules diffuse through a thin quiescent layer of solution adhering to the surface and into a well-mixed turbulent layer of solution (the environment). This diffusion process is described by Fick's First Law of Diffusion. According to this Law (14)

$$J = -D\left(\frac{\delta c}{\delta x}\right)_{y,z} \quad (1)$$

where D is the diffusion coefficient and J , the flux, is the quantity of substance diffusing per unit time through a unit area. Thus,

$$J = \frac{1}{A} \frac{dm}{dt} \quad (2)$$

Therefore,

$$\frac{dm}{dt} = -DA\left(\frac{\delta c}{\delta x}\right)_{y,z} \quad (3)$$

Let

$$\frac{(c - c_s)}{x} \approx \left(\frac{\delta c}{\delta x}\right)_{y,z} \quad (4)$$

where c is the concentration in the bulk solution, c_s is the organotin concentration at the coating surface, and δ is the thickness of the quiescent layer which is a function of the water velocity moving pass the surface. See Figure 1 for a pictorial representation of the leaching process. Thus,

$$\frac{dm}{dt} = - \frac{DA}{x} (c - c_s) \quad (5)$$

For an experimental container of volume v , one obtains

$$\frac{dc}{dt} = - \frac{DA}{xv} (c - c_s) \quad (6)$$

If an organotin compound is a liquid, it will mix thoroughly with its matrix forming a homogeneous system. If the organotin compound is a solid, it may dissolve somewhat in the matrix and solvent forming a low polymers of associated toxicant molecules, or it may remain as solid particles dispersed throughout the matrix (15,16). The regions of surface that are active in the leaching process will depend on the development of pores and channels through the matrix as the organotin compound and/or matrix dissolve and leach into the environment. If the area of the surface plane is A_o and if f is the fraction of the plane that is active, then

$$A = f A_o \quad (7)$$

and

$$\frac{dc}{dt} = - \frac{DfA_o}{xv} (c - c_s) \quad (8)$$

Integrating, one obtains

$$c = c_s - c_s \exp\left(- \frac{DfA_o}{xv} t\right) \quad (9)$$

The characterization of a coating involves determining c_s and Df/x by measuring the bulk concentrations at various times and using the least squares technique to fit the theoretical equation to the experimental data. The general form of the equation is

$$c = c_s - c_s \exp\left(- \alpha \frac{A_o}{v} t\right) \quad (10)$$

where α is Df/x and is characteristic of the coating for a given temperature and flow condition.

Now consider obtaining an expression for the lifetime of the coating knowing c_s and α :

$$\frac{dm}{dt} = -\frac{DfA_o}{x} (c - c_s) \quad (11)$$

In a large body of water (ocean or harbor), c is always negligible and A_o is the surface area of the ship continuously exposed to the water. Then

$$\frac{dm}{dt} = \alpha A_o c_s \quad (12)$$

Integrating, one obtains

$$m = \alpha A_o c_s t \quad (13)$$

where m is the mass of organotin leached out of the coating. Define a critical density, ρ_τ , which is achieved at the time τ where fouling becomes appreciable (say 50%). The mass of organotin lost from the coating is given by

$$m_\tau = \alpha A_o c_s \tau, \quad (14)$$

and

$$\rho_\tau = \rho_o - \alpha c_s \tau \quad (15)$$

where ρ_o is the original mass of organotin per unit area. The minimum useful lifetime of the coating is then given by

$$\tau = \frac{1}{\alpha c_s} (\rho_o - \rho_\tau) \quad (16)$$

Experimental Section

Static leach tests were performed on aluminum panels coated with Alum-A-Tox (a commercial coating obtained from Standard Paint and Varnish Company of New Orleans) containing an organotin compound as toxicant. Alum-A-Tox is a vinyl-type coating containing titanium dioxide as pigment. Either bis(tributyltin) oxide, tributyltin chloride, tributyltin acetate, triphenyltin acetate, or triphenyltin chloride was mixed into the coating so that a coating composition of 3.6% Sn by weight was obtained. The aluminum panels (3" x 4" x 1/16") were sanded and cleaned with carbon tetrachloride. They were sprayed with the paint on one side and were allowed to air-dry for at least three weeks. Each panel was then immersed in 550 ml artificial sea water (17) in a beaker and maintained at $21 \pm 2^\circ\text{C}$ for the duration of the experiment. Solutions were replaced at the time of sampling (about every two weeks). The leachates were analyzed for tin using colorimetric methods.

The organotin concentration in the leachate was determined

after extracting the organotin compounds from the aqueous phase. The 550 ml of leachate from a given sample were diluted to 1000 ml with distilled water, and 100 ml was then pipetted into a separatory funnel. After adding 10 ml of chloroform to the separatory funnel and shaking vigorously for one minute, the contents were allowed to separate into two layers. The chloroform layer was removed, the extraction procedure was repeated three to four times to achieve complete extraction of the organotin compounds, and all of the chloroform portions were combined. Tin present in the chloroform was determined by the dithizone colorimetric method (18).

Any tin species remaining in the aqueous phase were subjected to an oxidation procedure before measuring the tin concentration. The aqueous phase was acidified with 16M HNO_3 and evaporated cautiously to a small volume. After adding 5-6 ml of 18M H_2SO_4 , the solution was evaporated to the incipient fumes of SO_3 . The residue was dissolved in distilled water and the resulting solution was diluted to 100 ml. The tin present in this solution was determined using the phenylfluorone colorimetric method (19). No tin above background was found in the aqueous phase for any sample. This suggests that the tin in the leachate is solely in the organotin form and not in the inorganic form.

It was assumed that the leaching would be slow enough that

$$\frac{\Delta c}{\Delta t} \approx \frac{dc}{dt}$$

where Δc is the measured organotin concentration and Δt is the time of accumulation. The leach rate - time data were fitted with a quadratic equation, and this empirical equation was integrated to obtain concentration-time data. Initial guesses for c_s and α were obtained after comparing the concentration-time data with simulated data generated for trial values of c_s and α by a program called LEACH2. The best values of c_s and α resulting from the previous procedure were further refined by a least squares program called LEACH1. As the concentration-time data were obtained from smoothed data, uncertainties in c_s and α have not been calculated.

To determine the solubilities of the organotin compounds used in this study, 50 mg of each compound were mixed with 100 ml of artificial sea water. The solutions were maintained at $21 \pm 2^\circ\text{C}$ for two days with occasional stirring. The amount of organotin dissolved in the sea water was determined by the dithizone colorimetric method (18).

Results and Discussion

The leaching results for the tributyltin compounds and the triphenyltin compounds are shown in Figure 2 and Figure 3

respectively. The parameters giving the best fit to the concentration-time data and the solubilities are given in the Table.

TABLE I

Parameters for Alum-A-Tox Coatings Containing Organotin Compounds

Toxicant	c_s (ppm)	α (cm day ⁻¹)	Solubility (ppm)
$\phi_3\text{SnOAc}$	7.2	0.16	3.34
$\phi_3\text{SnCl}$	2.2	0.18	4.22
$(\text{C}_4\text{H}_9)_3\text{SnOAc}$	12.8	0.19	1.88
$(\text{C}_4\text{H}_9)_3\text{SnCl}$	3.3	0.45	1.57
$[(\text{C}_4\text{H}_9)_3\text{Sn}]_2\text{O}$	9.9	0.17	5.92

As t goes to large values, c approaches c_s (see equation 10). The maximum concentration of organotin in sea water is its solubility. From this argument c_s , the concentration of organotin at the surface, would have the solubility as its maximum value. From the table it can be seen that c_s is of the same order of magnitude as the solubility but the values of c_s are larger than the solubility in many cases.

The parameter α must be considered to be empirical. There are no diffusion constants available for organotin compounds, and it is impossible to measure the thickness of the quiescent layer. However, since the solvated organotin species are expected to be the same (for R_3SnX where R is constant) in all cases (10), the same diffusion constant would be applicable. Under constant flow conditions, the thickness of the quiescent layer would be the same for all the coatings. Values of α would therefore provide a relative measure to rank or order the fraction of the active surface between the various coatings. This fraction of the surface that is active in the diffusion process is expected to be dependent on the physical state of the organotin compound in the coating as well as the porosity of the matrix. However, since only one matrix and a few selected toxicant compounds were studied, any further discussion based on the magnitude of c_s and α would be entirely speculative.

These preliminary results are promising in that an empirical diffusion model has been developed which appears to be generally applicable for describing the leaching process associated with a series of organotin toxicants contained in a commercial coating vehicle. The results do indicate that it should be feasible to design laboratory testing procedures for making relative comparisons of the properties of a series of coatings of different formulations. However, further studies must be carried out before the optimum range of c_s and α for satisfactory antifouling performance can be ascertained. In addition, further testing is

necessary before additional refinements to the model can be made.

The experimental design for this initial study was not optimum in that the leaching rates were faster than anticipated. This makes suspect the assumption that the leach rate can be approximated by the measured organotin concentration divided by the accumulation time. The second iteration of this experiment will include other types of coating matrices and a new sampling procedure which will allow concentration-time data to be acquired directly. After these static tests have been completed, the effect of the depth of the quiescent layer and the possibility of accelerated leaching will be evaluated by carrying out all of the measurements on coatings mounted on rotating drums designed for a variety of speeds.

Acknowledgements

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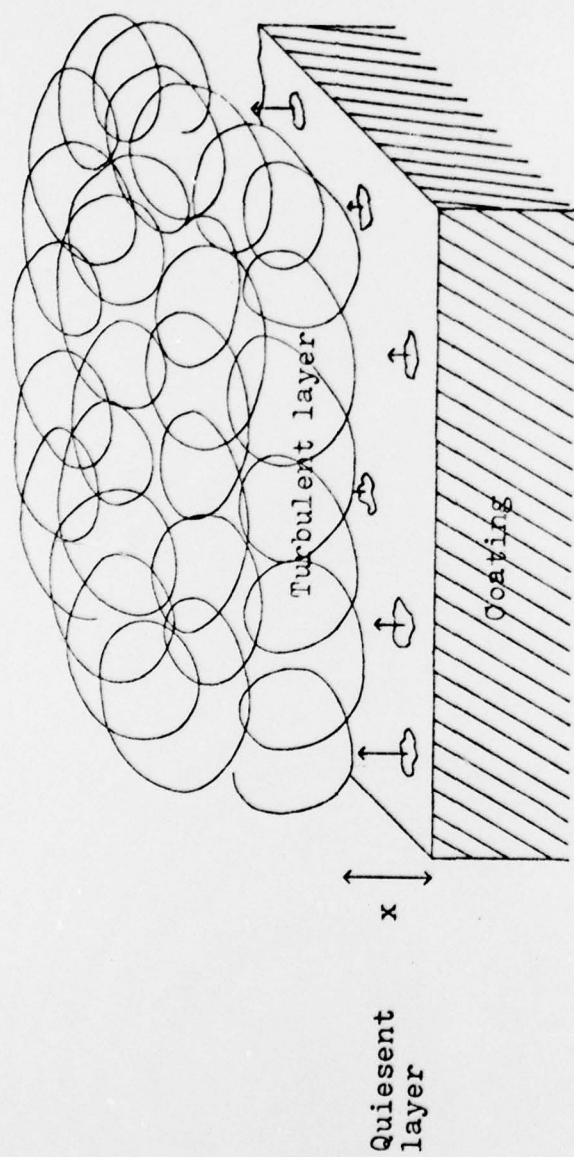
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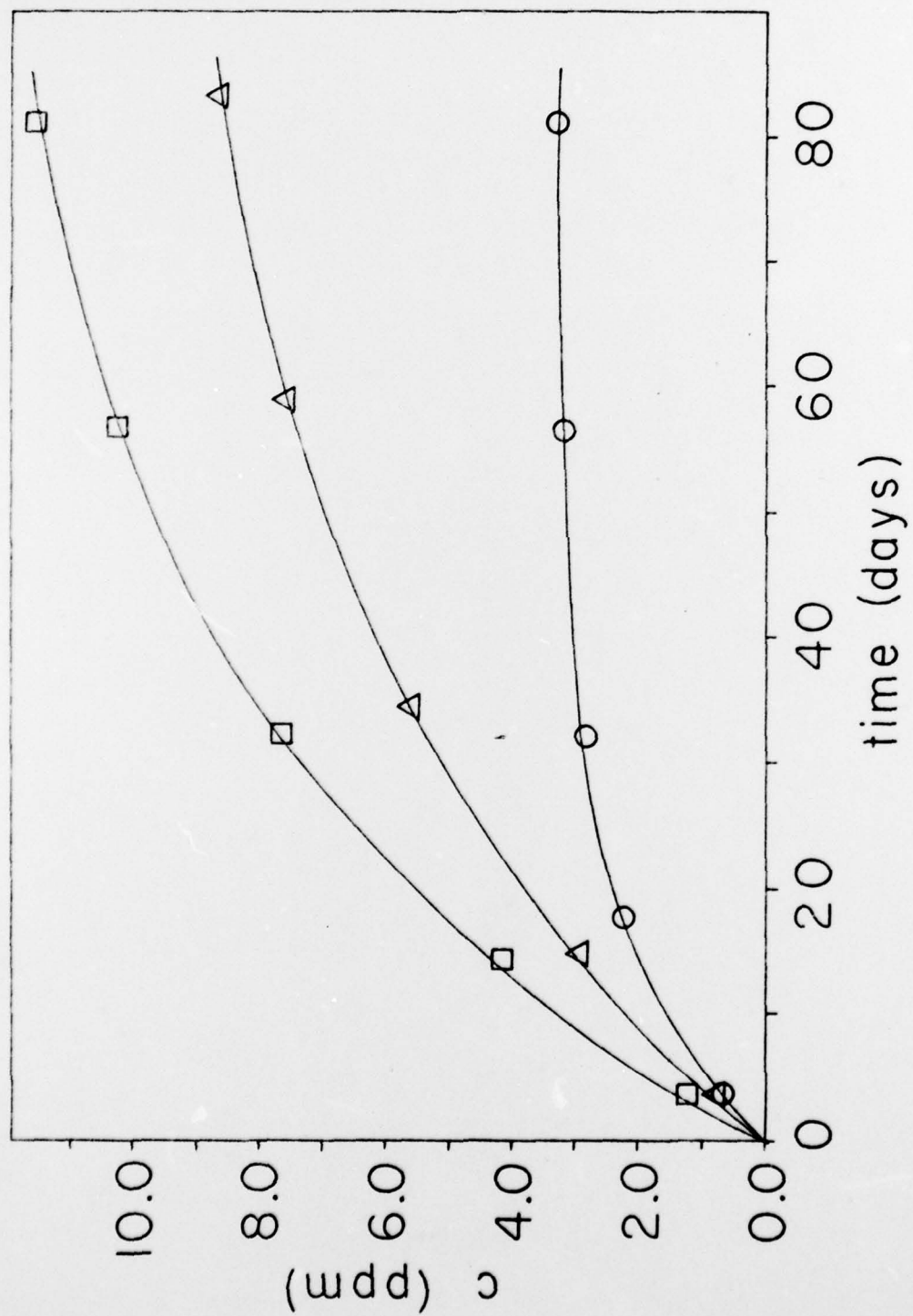
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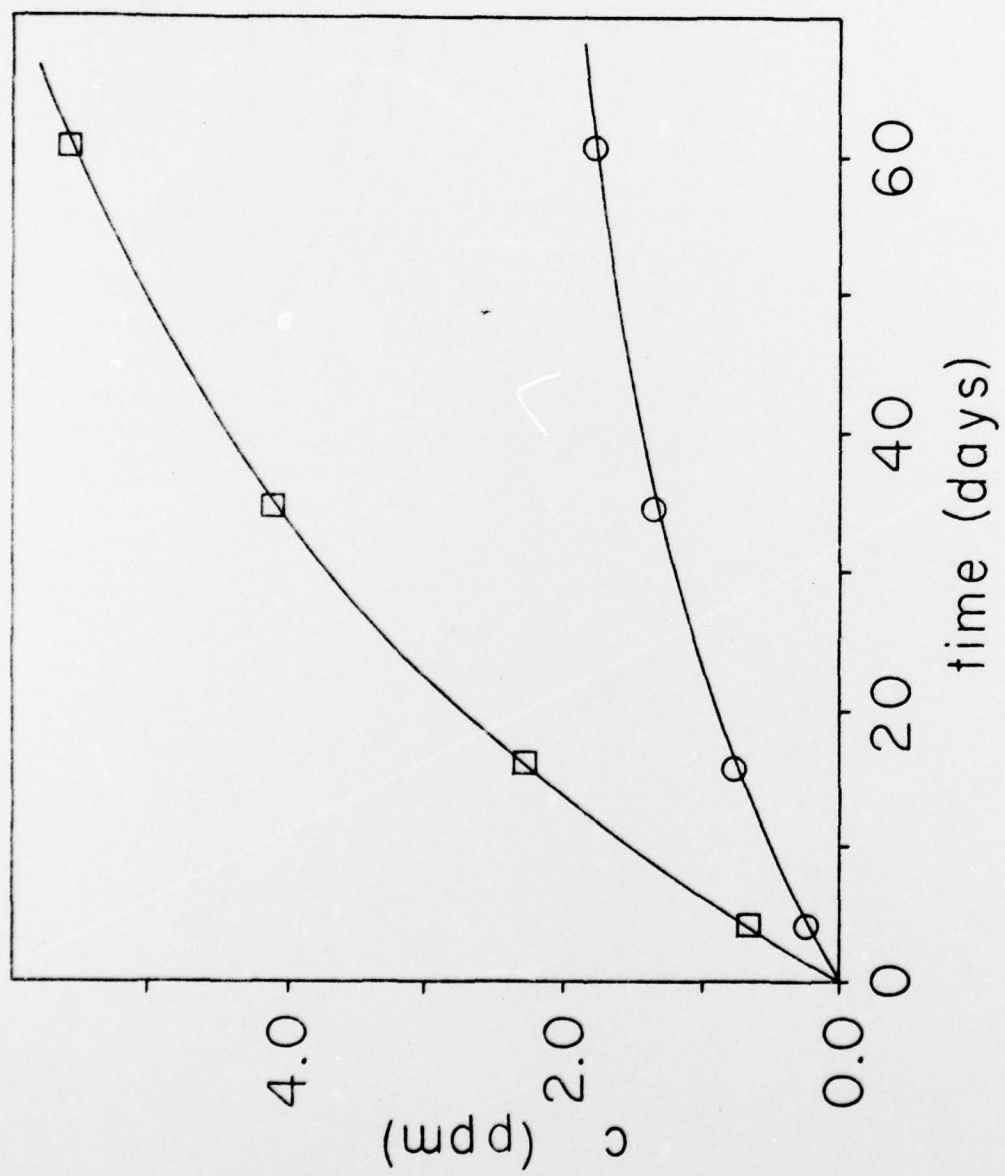
Figure 1. Pictorial representation of the leaching of toxicant molecules from an antifouling coating.

Figure 2. Leaching of tributyltin compounds from Alum-A-Tox coatings: \square is $(C_4H_9)_3SnOAc$, Δ is $[(C_4H_9)_3Sn]_2O$, and O is $(C_4H_9)_3SnCl$. Each solid line represents the least squares fit to equation (10).

Figure 3. Leaching of triphenyltin compounds from Alum-A-Tox coatings: \square is ϕ_3SnOAc and O is ϕ_3SnCl . Each solid line represents the least squares fit to equation (10).







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